Research Article

Fabrication of polypropylene/lignin blend sponges via thermally induced phase separation for the removal of oil from contaminated water

Abeer Alassod[1,2] · Syed Rashedul Islam[1] · Amjad Farooq[1] · Guangbiao Xu[1]

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Abstract
Polypropylene is widely used in oil spillage cleanup due to excellent characteristics. However, polypropylene is not renewable and biodegradable, which is unacceptable to introduce new pollutants while solving environmental disasters. Therefore, there is a high demand to explore a low-cost, environmentally friendly, and renewable technique for the fabrication of porous materials. In this work, lignin was chosen as the second blend in the polymer matrix. Polypropylene and renewable lignin sponges were successfully prepared using simple, inexpensive, controllable, scalable, and an environment-friendly method named thermally induced phase separation (TIPS). The surface morphology of obtained sponges was investigated using FTIR and SEM. FTIR analysis indicated that PP and lignin were physically blended. SEM analysis observed an interconnected porous network that acts as a capture site of oil, and lignin merged into PP. The contact angle of PP, PP5L, PP10L, PP15L, and PP20L was found to be 127.4°, 118.71°, 113.89°, 109.45°, and 107°, respectively. Furthermore, polypropylene/lignin sponges have good adsorption ability toward oils compared to polypropylene itself. The research detected that the highest oil sorption tests exhibited by PP10L sponge, which could absorb 983% of soybean oil, 788% of engine oil, and 550% of lubricating oil in the oil system, with high oil retention more than 90% after 24-h dripping. Besides, the results revealed that temperature has a significant effect on oil absorption. All of these features make polypropylene/lignin blend sponges promising sorbents for the oil spills cleanup, not only for oil recovery but also helps in cleaning the environment.

Keywords Polypropylene · Lignin · Oil sorption · Thermally induced phase separation · Thermal stability

1 Introduction

Oil is a part of the natural environment for many decades and is a naturally occurring substance with the rapid development of oil production, transportation, exploration, and storage [1–4]. The risk of oil spillage increased with the potential to create a notable environmental problem and also a great waste of energy [5–8]. Therefore, many methods have adopted to clean and recycle spilled oil. Among these methods, mechanical extraction by sorption materials is considered one of the most popular methods [9, 10]. An ideal oil sorbent material should have hydrophobicity, oleophilicity, sorption capacity, buoyancy, good oil–water selectivity, and reusability [11, 12]. At present, synthetic polymer materials, such as PP, have been widely used as oil sorbents due to unique features such as excellent mechanical and chemical properties as well as the ease of processing; density is less than that of water [13]. Further, PP has hydrophobic and oleophilic features, which make it potentially promising for materials for oil

✉ Guangbiao Xu, Guangbiao_xu@dhu.edu.cn | [1]Key Laboratory of Textile Science and Technology of Ministry of Education, College of Textiles, Donghua University, Shanghai 201620, China. [2]Department of Textile Engineering, Mechanical and Electrical Engineering University, Damascus, Syria.

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spill cleanup [14–16]. Nevertheless, these materials have some limitations that degrade very slowly, and that is considered to be mainly responsible for the phenomenon known as white pollution, which results in the accumulation of plastics on earth [17].

Moreover, the current price of PP has been estimated to be around $1210/ton [18]. Therefore, partial replacement of PP with low-cost, environmentally friendly, and renewable materials was the demand to use renewable sources for bio-economic perspective consideration. Recently, great attention to the development of renewable resources based on natural biomass sources has been an ever-growing field over the last decade [19]. Biocomposite is derived from biomass such as lignin, starch, and natural cellulose, which has been the subject of many studies due to the advantages such as economic and environmental factors [20, 21]. Among natural abundant and biomass materials, lignin is the second most abundant natural, simple biopolymer on earth after cellulose, which is still considered a by-product and burns as a low-value-added fuel [22, 23]. Currently, around 50 million tons of lignin is produced annually as a by-product of the pulp-and-paper industry. Only a small fraction (about 2%) is effectively utilized, and most of the lignin is burned or discharged into rivers, thereby causing serious environmental pollution and grievous waste of renewable resources [24, 25]. Lignin gained considerable attention for various applications. Chemically, lignin is a complex, three-dimensional amorphous polymeric material with aliphatic and aromatic subunits. Aromatic groups impart a polar character to polymers and contribute to their incompatibility with nonpolar polymers such as PP [26], polyethylene (PE) [27, 28], and polyester (PS) [29].

Many authors investigated the field of PP/lignin systems in the past [17, 30–32]. The treatment of oily wastewater is still a big task and also indispensable to developed efficient large-scale and economical methods to fabricate sorbents from low-cost materials. Hence, to solve the problems of high-cost devices and long production time, phase separation of the polymer solution is one of the useful methods to prepare porous polymers with interconnected porous structure. Several methods to induce phase separation are adopted; thermally induced phase separation method (TIPS) is one among those methods [33–36]. TIPS method is considered as a simple and clean process that also surpasses the extra effort needed for the removal of the template molecules. Another attractive feature of the TIPS method over other conventional techniques is the formation of an intrinsically interconnected porous network, that is controllable, versatile, convenient, and scalable [37].

Herein, this study aims to develop low-cost and environmentally friendly sponges prepared using lignin and PP via using thermally induced phase separation (TIPS). To best of the knowledge, using lignin incorporated in PP via thermally induced phase separation (TIPS) has not been reported yet. The outcomes from this research are believed to provide an essential reference in the practical application of polypropylene and lignin blend sponges in oil spill management.

Table 1 Properties of the test oils

Oil	Soybean oil	Engine oil (15 W-40)	Lubricating oil
Viscosity (mPa s)	65.3	234.5	21.70
Density (g cm^{-3})	0.92	0.87	0.82
Surface tension (mN m^{-1})	33.41	30.78	28.11

Table 2 Produced composites and their sample codes

Sample name	PP %	Lignin %
PP	100	0
PP5L	95	5
PP10L	90	10
PP15L	85	15
PP20 L	80	20

2 Materials and methods

2.1 Materials

PP pellets (PP), lignin (L), acetone, decalin, and 1-butanol were purchased from Aladdin, Shanghai, PR China. Three different oils, namely engine oil, soybean oil, and lubricating oil, were used. The densities and surface tensions of those oils were examined using dynamic contact angle tester (DCAT1), and ASNB2 Digital Rotary Viscometer tested the viscosities. Every test was repeated three times to obtain an average value. The test temperature was maintained at 22 ± 2 °C. The properties of the studied oils are reported in Table 1.

2.2 Fabrication of PP/lignin blend sponges

The fabrication of polypropylene/lignin blend sponges by the thermally induced phase separation technique (TIPS) was accomplished as follows. Polypropylene pellet was dissolved in the mixed solvent of decalin and 1-butanol at 115 °C, and lignin was added manually in solution. Then, the solution was cooled at 20 °C, resulting in the phase separation of the polymer in the form of a sponge. The sponge was immersed with acetone and subsequently dried under vacuum. The addition levels of dried lignin were set at 0, 5, 10, 15, and 20 wt% of the total weight of polypropylene pellets, as reported in Table 2. The general protocol for the procedure for the fabrication of sponge is illustrated in Fig. 1.

2.3 Characterization

2.3.1 Morphology

A scanning electron microscope of polypropylene/lignin blend sponges' surface was characterized by using scanning electron microscopy (SEM) (FLEX SEM1000, Hitachi, Japan). For SEM observation, samples were cut, then fixed on double tape, and after that plated with a thin film of gold before measurement.

2.3.2 Fourier transform infrared measurement

The infrared spectrum was performed using the FTIR spectrometer (Nicolet 6700, Thermo Fisher, USA). The FTIR spectra were recorded in the range 400–4000 cm^{-1} wavenumber.

2.3.3 The oil absorption experiment

The absorption ability was studied by weighing the samples before (W_i) and after (W_t) immersing them in oils (soybean, engine) as percent weight gain (Q) [38, 39]. Samples were lifted and placed with free oil dripping out for 24 h, weight was measured (W_d), then the retention was calculated (R) [40], and the weight gain and retention were calculated according to Eqs. (1) and (2):

$$Q = \frac{W_t - W_i}{W_i} \times 100 \tag{1}$$

$$R = \frac{W_{d-W_i}}{W_t - W_i} \times 100. \tag{2}$$

The ability to reuse the sorbents for oil sorption was studied using soybean oil, the sorbents with oil were squeezed, and then squeezed sorbent was again used with the same procedure as described above. The sorption—squeezing process was repeated many times under identical conditions to evaluate the reusability of the sorbent.

3 Results and discussion

3.1 Morphology

In all tested samples, the three-dimensional interconnected porous structures were investigated by SEM, as depicted in Fig. 2. This is explained to the phase separation of the polymer solution during the cooling process, in which polymer-rich regions contributed to the formation of skeletons. The porous structures provided sufficiently storage space to capture the oil. The pore volume decreased by squeezing the sorbent, causing the recovery of the absorbed oil. The figure revealed the characterization of blend sponge that lignin achieved incorporation in the PP blend successfully [41]. Moreover, the introduction of lignin with three-dimensional structures effectively supported the micro-/nanoscale structures [42]. It is clearly obvious that pure polypropylene had a smooth surface, as shown in Fig. 2a, while with addition, lignin assembles a rough surface, as shown in Fig. 2b–e, which indicates the small lignin particles embedded in polypropylene matrix successfully [41].

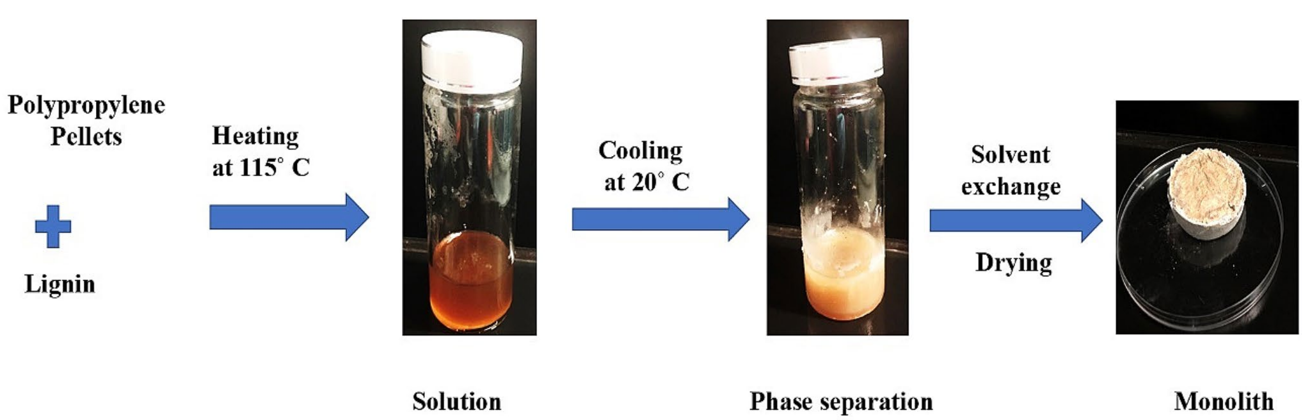

Fig. 1 A general protocol for the preparation of polypropylene/lignin blend sponges by a thermally induced phase separation technique

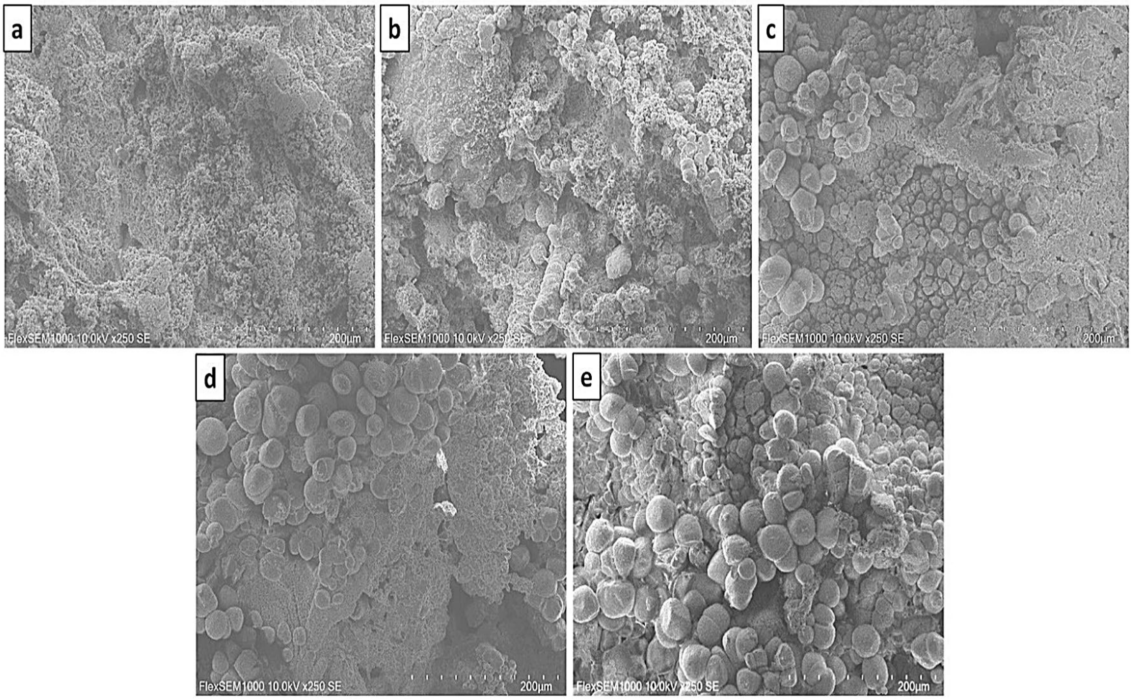

Fig. 2 SEM image **a** PP, **b** PP5L, **c** PP10L, **d** PP15L, **e** PP20L

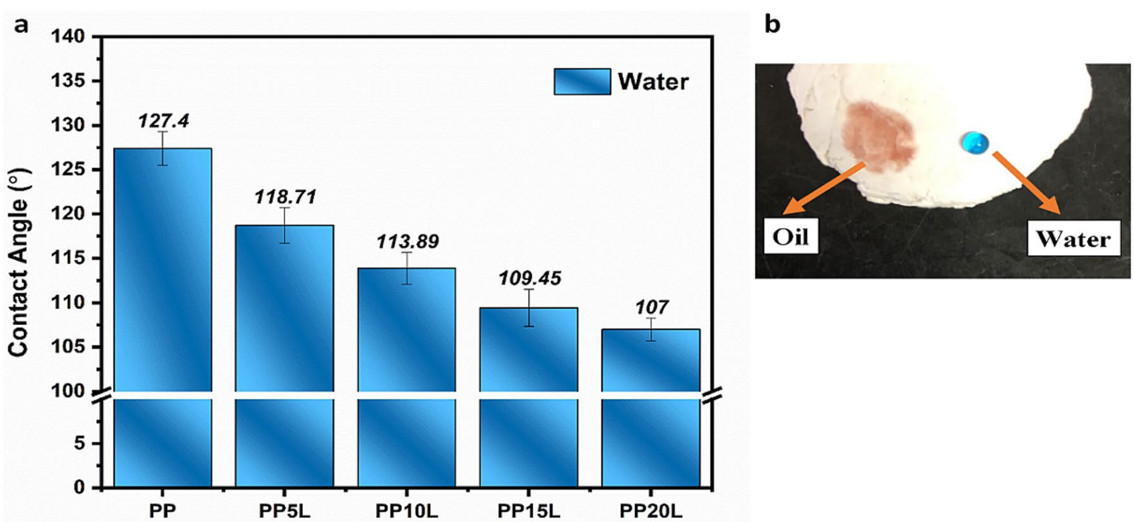

Fig. 3 **a** Water contact angle of polypropylene and polypropylene/lignin blend sponges, **b** Surface wettability of blend sorbent. Engine oil (dyed in red) and water (dyed in blue with methylene blue) were spotted on the surface of the sorbent

3.2 Hydrophobicity and lipophilic

The wettability "contact angle result" of polypropylene and polypropylene/lignin was performed to study the behavior of different lignin content in the polypropylene matrix, as displayed in Fig. 3a. PP showed a hydrophobic feature with a contact angle around 127.40°, while the addition of 5 wt% of lignin in polymer matrix reduced contact angle for value around 118.71°. The obtained results may be attributed to the contribution of the several free polar groups from lignin [43]. With increasing lignin, loading in the PP matrix to values above 10 wt% provides contact angles for PP10L, PP15L, and PP20L, which were 113.89°, 109.45°, and 107°, respectively. However, the contact angle for these sorbents is more than 90° according to the standard definition of hydrophobic materials [44]. These sorbents also have hydrophobic features. On the another side to indicate lipophilic feature for sorbents, it was obviously noticed that tested sorbents immediately absorbed oil droplets, as shown in Fig. 3b.

3.3 Fourier transform infrared spectroscopy analysis

Fourier transform infrared (FTIR) measurement of polypropylene, lignin, and their composites polypropylene/lignin with different proportions between wavenumbers 4000 and 500 cm^{-1} is shown in Fig. 4. FTIR spectra of lignin showed the wide band in the range between 3400 and 3500 cm^{-1} due to the hydroxyl groups (O–H stretching) [45], following a range of bands around 2936 cm^{-1} (C–H stretching) and 2846 cm^{-1} (C–H tensor). One of the essential peaks in all polypropylene/lignin is the band at a range between 3400 and 3500 cm^{-1}, which becomes clearly observed when increasing the addition of lignin in polypropylene matrix (PP20L > PP15L > PP10L > PP5L). Furthermore, from this figure it can be seen that PP had peaks at 2950 cm^{-1}, 2916 cm^{-1}, 2866 cm^{-1}, and 2836 cm^{-1}, which represented (C–H stretching); also two bands are observed at 1373 cm^{-1} (syringyl group) and 1360 cm^{-1} ($C–H_3$ bonding and C=O stretch) in polypropylene/lignin structure like polypropylene [46].

It can be observed from a typical FTIR spectrum of added lignin in polypropylene that the composition of the bonds did change but increased the intensity of the peaks. Other important structures in lignin are aromatic rings because aromatic skeleton vibrations at peaks 1597 cm^{-1} and 1510 cm^{-1} are visible PP/lignin [47]. Also, the presence of new bands around 1710 cm^{-1} in the carbonyl region can be associated with conjugated carbonyl stretching [48]. Lignin was detected at 1250 cm^{-1}, 1210 cm^{-1}, 1115 cm^{-1}, and 1037 cm^{-1} corresponding to syringyl absorptions, guaiacyl ring breathing, which was observed in the spectrum of polypropylene/lignin (Fig. 4).

3.4 Oil sorption

Polypropylene/lignin blend sponges were prepared via the TIPS method that are promising sorbents for oil cleanup because of their 3-D interconnected macroporous structure, hydrophobicity, super-oleophilicity, and also because incorporated lignin provides micro-/nanoscale structures, respectively, as discussed above [42]. In this case, it was observed through the experimental test that the adsorption process speed is slower, which indicates the presence of large holes and small macropores. That increase in absorbed oil into a tested sample is a vital key for the fast removal of spilled oils. The weight gain of tested samples in the oil system and oil/water system was measured, as shown in Fig. 5a. The weight gain of PP, PP5L, PP10L, PP15L, and PP20L for soybean oil is 480%, 685%, 983%, 875%, and 788%, respectively. The engine oil weight gain of these sorbents indicates a similar tendency that is 388%, 543%, 788%, 701%, and 645%, respectively. Besides, lubricating oil weight gain of these sorbents indicates a similar tendency that is 275%, 320%, 550%, 451%, and 385%, respectively. Figure 5b, c, and e shows the weight gain of sorbents in the oil–water system. It was observed that

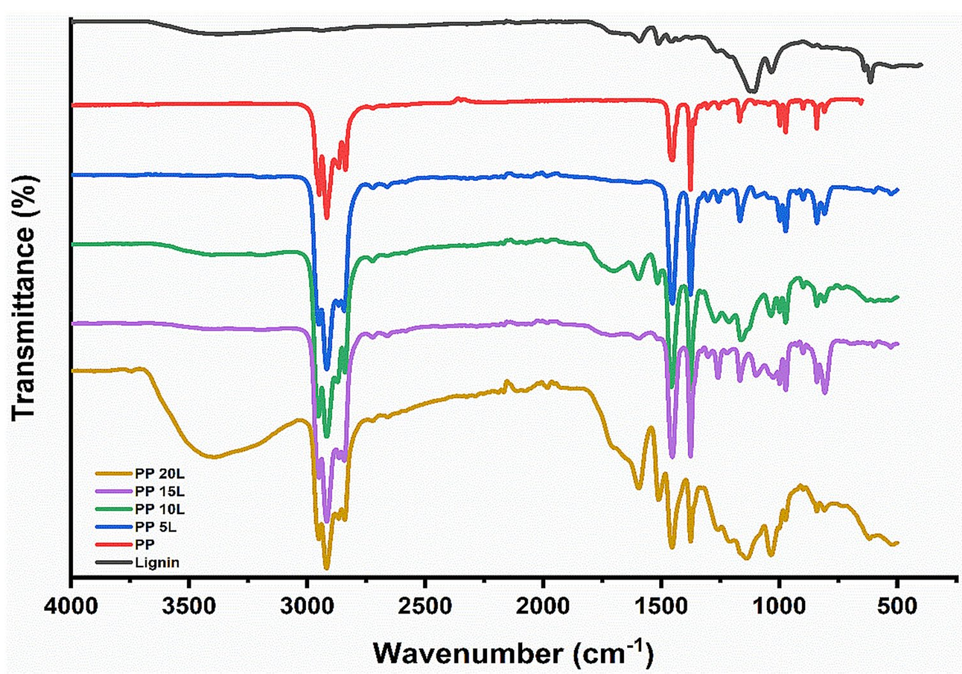

Fig. 4 FTIR spectra of polypropylene, lignin, and polypropylene/lignin blend sponges

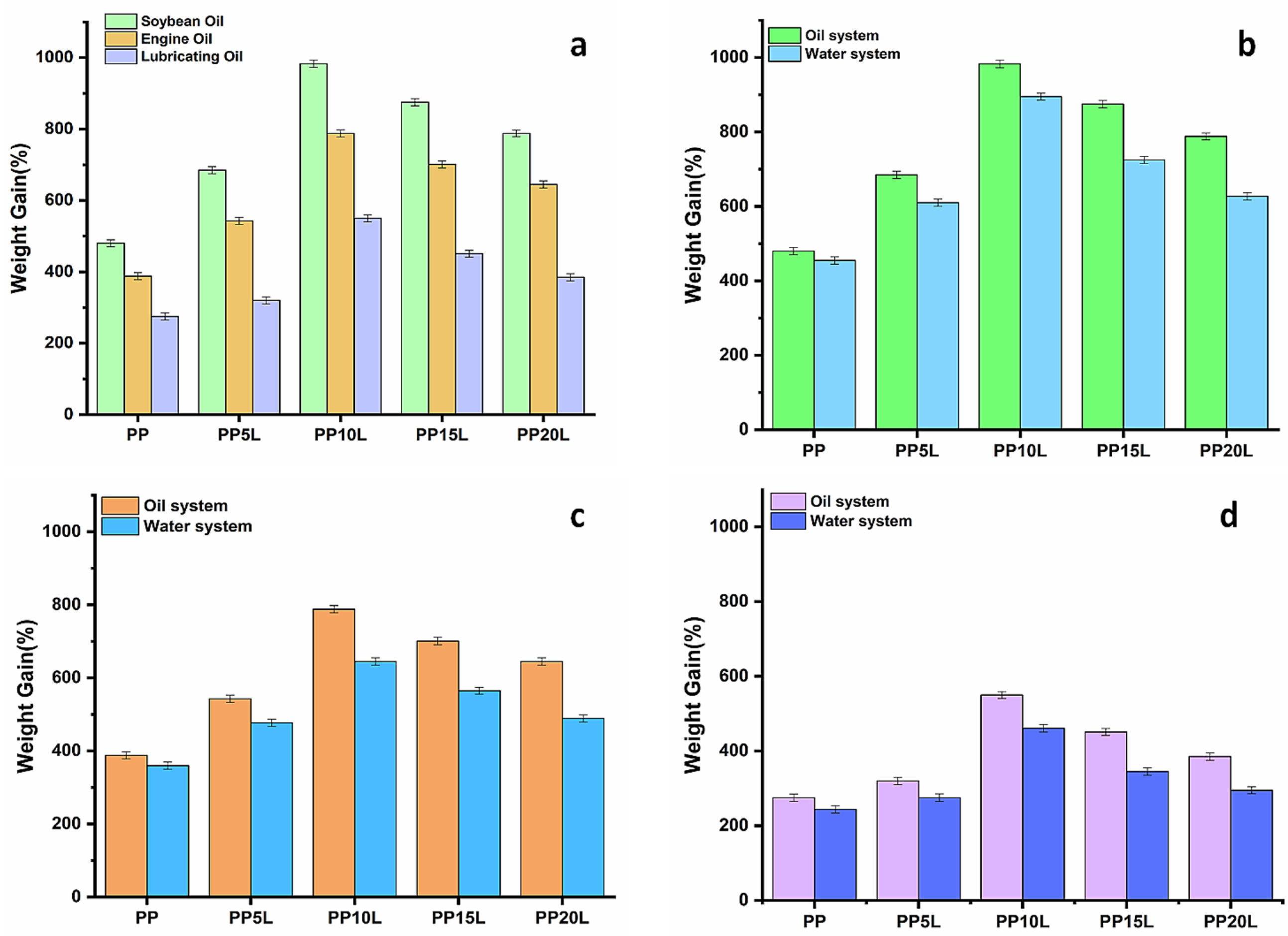

Fig. 5 Absorption capacities of polypropylene and polypropylene/lignin blend sponge **a** oil system, **b** soybean oil system/water system, **c** engine oil system/water system, **d** lubricating oil system/water system

weight gain for soybean oil is 455%, 610%, 895%, 725%, and 627%, respectively, for engine oil; the tendency is the same behavior, that is 360%, 477%, 645%, 565%, and 489%, respectively. And for lubricating oil, the tendency is the same behavior, that is 240%, 275%, 461%, 345%, and 295%, respectively. All obtained results showed higher oil sorption in the oil system when compared to the oil/water system. It was observed with the increased value of lignin more than 10 wt%, and there was a slight reduction in oil sorption. This result corroborates the contact angle results. Even that still PP15L and PP20L showed an improvement in sorption capacity compared to the blank one for both systems, it was seen that there is an increase in oil sorption with increased addition of lignin content. All polymer modified by lignin had to show the positive impact of lignin on oil sorption in the system. Lignin slightly reduced the hydrophobic character of the polymer matrix due to the polar groups in the structure. Still, it also has aromatic groups obtained from benzene, which can favor the sorption of organic compounds [49].

High oil retention ability is an essential feature to keep oil encapsulated in the sorbent so that the sorbent can be relocated from the water to a nominated area without losing the oil into the surrounding so that it avoids the second contaminant. The oil retention after 24 h dripping for tested sorbents was measured as the values listed in Table 3. The oil retention for PP, PP5L, PP10L, PP15L, and PP20L for soybean oil was 78.57%, 88.04%, 92.20%, 90.96%, and 89.11%, respectively. The engine oil retention

Table 3 Oil retention of PP and PP/lignin blend sponges

Sample code	Soybean oil retention (%)	Engine oil retention (%)	Lubricating oil retention (%)
PP	78.57 (1.34)	82.97 (1.77)	72.70 (1.93)
PP5L	88.04 (1.47)	88.92 (1.89)	75 (1.87)
PP10L	92.20 (1.17)	93.87 (1.32)	90.90 (1.39)
PP15L	90.96 (1.34)	91.61 (1.92)	83.30 (1.77)
PP20L	89.11 (1.56)	89.89 (1.45)	78.94 (1.71)

of these sorbents indicates a similar tendency, that is 82.97%, 88.92%, 93.87%, 91.61%, and 89.89%, respectively. And lubricating oil retention of these sorbents indicates a similar tendency, that is 72.70%, 75%, 90.90%, 83.30%, and 78.94%, respectively. Generally speaking, it was noted that tested samples absorbed a higher amount of soybean oil than the engine oil and lubricating oil. This could be attributed to the fact that soybean oil is heavier than engine oil within the same unit volume. On the other hand, soybean oil was more ready to drip out from tested sorbents than engine oil. The draining takes place when the capillary pressure is insufficient to capture the weight of oils. The heavy nature of soybean oil combined with its lower viscosity than engine oil was favorable for the dipping process [50].

Figure 6a shows a mixture of oil and water, in which the colorless transparent part is water and the colored part is oil. As shown in Fig. 6b, the blend material floats on the

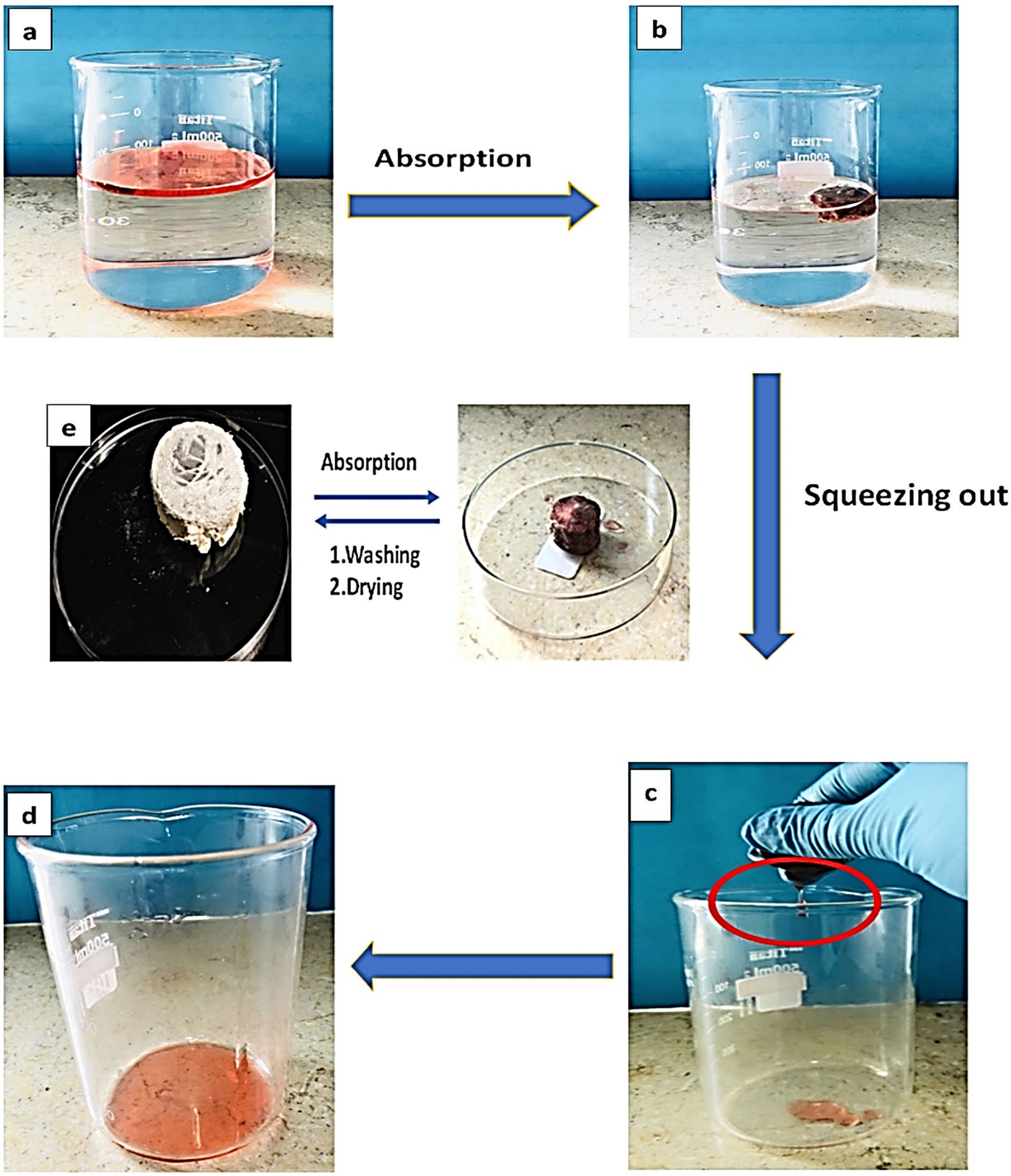

Fig. 6 The absorption and recycling process of soybean oil (oil dyed with red color) and the recovery of the PP 10L sponge by washing and drying in the air

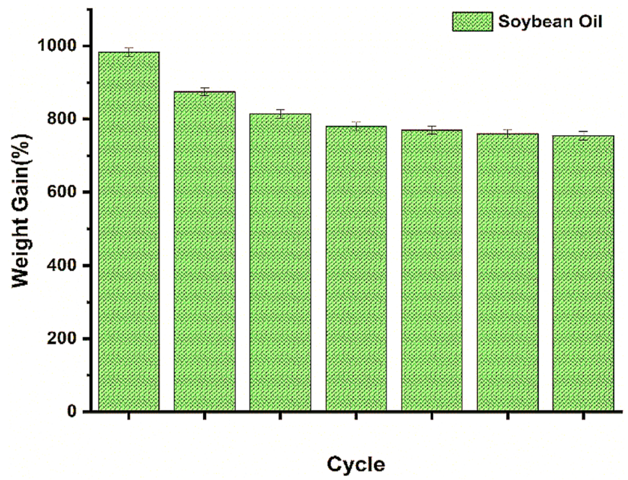

Fig. 7 Absorption recyclability of PP 10L sponge toward soybean oil

surface of the water. Once contacted, the floating oil drop is quickly and selectively collected inside the blend material. The adsorbed oil gathered easily by hand squeezing, as shown in Fig. 6c. After repeating the process several times in water and oil, the oil was successfully separated. The sponge could be easily reused by washing and dried subsequently, as shown in Fig. 7, which considers valuable property for practical applications. As described above, the excellent absorption properties make sponge a recyclable oil sorbent for large-scale oil spill cleanup.

Temperature is a significant parameter in the oily wastewater research since the temperature differs due to areas and seasons [51]. In this study, the different temperature was taken in the range 22 °C–35 °C–45 °C–55 °C and 65 °C, respectively. The results of the experiments were presented, as shown in Fig. 8. It was shown the oil temperature effect on the weight gain of tested samples. It was observed that with an increase in oil temperature

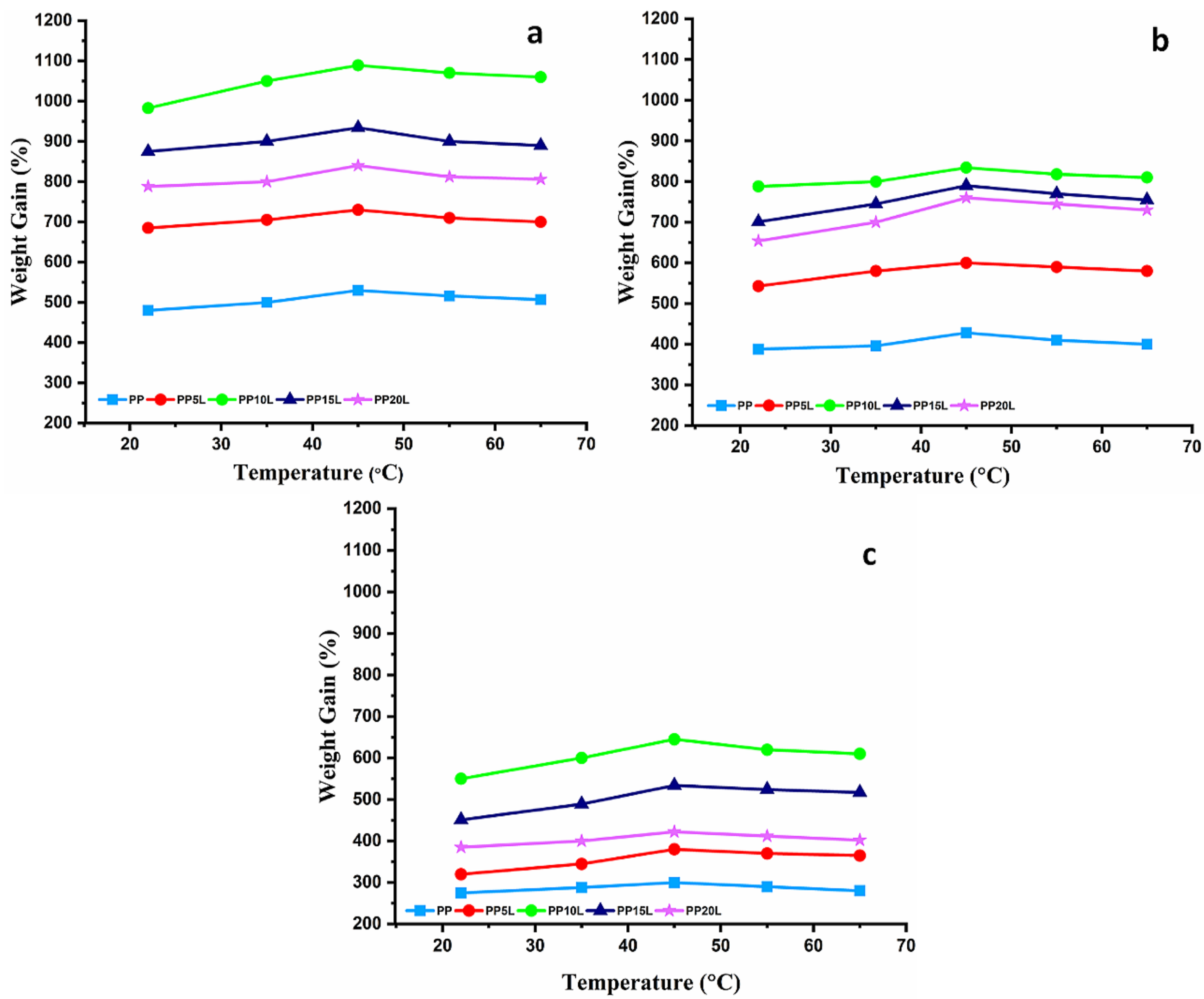

Fig. 8 Effect of temperature on oil absorption capacities of sponges **a** soybean oil, **b** engine oil, **c** lubricating oil

from 22 °C until reaching temperature 45 °C, the oil uptake (weight gain) increased gradually. This indicates that oil viscosity is directly related to the temperature, which can be explained to the random motion of particles increase, which supports the opportunity of oil droplets to be attached on the sorbent surface and penetrate the pores. Furthermore, with increasing temperature beyond 45 °C, the weight gain of tested samples decreases, and oil will be released from the pores and surface of the sorbent due to oil [52].

4 Conclusion

In this study, novel porous sponges based on economically and commercially available PP and lignin were successfully fabricated using the low-cost, simple process, eco-friendly, so-called thermally induced phase separation method (TIPS). Phase separation using the TIPS method was achieved in a short time, which shows a great advantage for the cleanup of a sudden oil spill accident. SEM showed sponges with a three-dimensional interconnected porous structure and showed lignin mixed with polypropylene. FTIR analysis revealed the successful blend of polypropylene and lignin together. Sponges showed good adsorption ability to oils. The absorbed oils were easily recycled by squeezing the sorbents manually. Moreover, the weight gain (%) of tested samples was found dependent on the temperature of the media. Considering oil absorbency of sponge, as well as the template-free and versatile fabrication method, this research presented a method for the design and fabrication of blend porous materials from polypropylene and lignin.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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